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METHODS OF CHEMICAL ANALYSIS OF SILICO-CHROMIUM

PART I ANALYSIS OF SILICON AND CHROMIUM

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SILICO-CHROMIUM

PART I ANALYSIS OF SILICON AND CHROMIUM

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SILICO-CHROMIUM

PART I ANALYSIS OF SILICON AND CHROMIUM

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 14 June 1968, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Silico-chromium is used in the production of chromium bearing alloy steel requiring simultaneous addition of silicon and chromium. This part deals with the analysis of the main alloying elements silicon and chromium. Analysis of aluminium and impurities will be covered in subsequent parts.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the ' 1967 Book of ASTM Methods for chemical analysis of metals: Parts 32 ' issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes methods for the analysis of silicon and chromium in silico-chromium in the ranges specified in IS : 2024-1962†.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 1472 (Part II)-1962‡.

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*Rules for rounding off numerical values (*revised*).
†Specification for silico-chromium.
‡Methods of sampling ferro alloy, Part II.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. USE OF FILTER PAPERS

4.1 In some of the methods prescribed in this standard, relative numbers of Whatman filter papers, which are commonly used, have been specified. However, any other suitable brand of filter papers of corresponding porosity may also be used.

5. DETERMINATION OF CHROMIUM BY THE PEROXIDE FUSION (VOLUMETRIC) METHOD

5.1 Outline of the Method — The sample is decomposed by sodium peroxide fusion and chromium in the solution of the sample is reduced by measured amount of ferrous ammonium sulphate solution. The excess of the latter is titrated with standard dichromate solution.

5.2 Apparatus — Nickel crucible (30-ml capacity) and nickel rods.

5.3 Reagents

5.3.1 *Sodium Peroxide* — solid.

5.3.2 *Dilute Sulphuric Acid* — 1 : 4 (*v/v*).

5.3.3 *Potassium Permanganate Solution* — 2 percent (*w/v*).

5.3.4 *Manganese Sulphate Solution* — 10 percent (*w/v*).

5.3.5 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962†).

5.3.6 *Standard Ferrous Ammonium Sulphate Solution* — 0·2 N. Dissolve 78·4 g of pure ferrous ammonium sulphate crystals [$FeSO_4 . (NH_4)_2 SO_4 . 6 H_2O$] in cold sulphuric acid (1 : 19) and make up to 1-litre with this acid. Standardize against standard potassium dichromate solution (0·2 N).

5.3.7 *Phosphoric Acid* — 85 percent.

5.3.8 *Standard Potassium Dichromate Solution* — 0·2 N. Dry pure potassium dichromate (AR) in an oven at 105° to 110°C for one and a half hours, cool in a desiccator and transfer carefully through a funnel

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*Specification for water, distilled quality (*revised*).

†Specification for hydrochloric acid (*revised*).

exactly 9·808 g to a 1-litre volumetric flask using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

5.3.9 *Sodium Diphenylamine Sulphonate Indicator Solution* — Dissolve 0·2 g of sodium diphenylamine sulphonate in 100 ml of hot water.

5.4 Procedure

5.4.1 Take about 0·5 g of the accurately weighed sample in a 50-ml nickel crucible. Add about 5 g of sodium peroxide and mix the contents thoroughly with a nickel rod and carefully clean the rod of adhering particles by scraping with another rod. Cover the mixture with a layer of about 1 g of sodium peroxide. Carry out the fusion carefully till the contents have melted down without spattering. Rotate the crucible carefully to stir any unattacked particles in the bottom and sides maintaining it at a low red heat. Just before completion of the fusion, increase the temperature of the crucible to bright redness for one minute and then allow to cool to room temperature.

5.4.2 Transfer the cold crucible containing the melt, along with the lid, to a covered 600-ml beaker containing 250 ml of water. After the reaction has subsided, remove the crucible and lid and rinse them thoroughly with water receiving all the washing in the same beaker. Boil the contents of the beaker briskly for 10 minutes, cool, treat with dilute sulphuric acid to 50 ml excess and raise the temperature to boiling. Treat with sufficient potassium permanganate solution to cause a definite pink coloration. Boil for 5 minutes and add 5 ml of manganese sulphate solution and boil for a further 5 minutes. Add a few drops of concentrated hydrochloric acid and boil. Filter, if necessary.

5.4.3 Cool to room temperature and run in a measured excess of standard ferrous ammonium sulphate solution (1·25 ml of 0·2 N solution is roughly equivalent to one percent chromium on a 0·5 g sample). The point, at which reduction is complete, may be detected by the change in the deep green colour that is developed. Add 5 ml of phosphoric acid and titrate the excess ferrous ammonium sulphate with standard potassium dichromate solution using sodium diphenylamine sulphonate as indicator.

5.4.4 Make a blank determination following the same procedure and using the same amount of all the reagents.

5.5 Calculation

$$\text{Chromium, percent} = \frac{(A - B)\, C \times 1{\cdot}734}{D}$$

where

A = volume in ml of the standard potassium dichromate solution required for the blank,

B = volume in ml of the standard potassium dichromate solution required for the excess ferrous ammonium sulphate,

C = normality of the standard potassium dichromate solution used, and

D = weight in g of the sample taken.

6. DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD

6.1 Outline of the Method — The sample is fused with sodium peroxide and the fused mass evaporated with hydrochloric acid, baked and then silicon estimated by hydrofluorization as usual.

6.2 Apparatus — Nickel crucible 50-ml capacity and nickel rods.

6.3 Reagents

6.3.1 *Sodium Peroxide* — solid.

6.3.2 *Dilute Hydrochloric Acid* — 1 : 1 and 1 : 20 (*v*/*v*).

6.3.3 *Dilute Sulphuric Acid* — 1 : 1 (*v*/*v*).

6.3.4 *Hydrofluoric Acid* — 40 percent.

6.4 Procedure

6.4.1 Fuse 0·5 g of an accurately weighed sample with sodium peroxide by following the procedure given under **5.4.1**. Extract the melt with minimum quantity of hot water in a covered porcelain washing the crucible and lid with jet of hot water. Acidify carefully with dilute hydrochloric acid (1 : 1) and add about 50 ml excess. Evaporate to dryness and bake for one hour at 100° to 110°C. Redissolve the mass in 40 ml of dilute hydrochloric acid (1 : 1) by warming and dilute with 150 ml of hot water. Raise to boil, allow to settle slightly and filter through filter paper No. 40. Wash 10 to 12 times alternately with hot dilute hydrochloric acid (1 : 20) and hot water. Finally rinse with hot water till the washings are free from chlorides. Preserve the paper and the residue (*A*).

6.4.2 Evaporate the filtrate and washings to dryness and repeat the procedure of baking, washing, etc, as in **6.4.1**. Preserve the paper and residue (*B*).

6.4.3 Place the papers and residue (A) and (B) in the platinum crucible and dry completely on a hot-plate. Ignite the crucible over Bunsen flame carefully and finally heat in a muffle furnace at about 1 050°C for 30 minutes, cool in a desiccator and weigh. Add sufficient dilute sulphuric acid to moisten the residue, then carefully about 10 ml of hydrofluoric acid and cautiously evaporate to dryness. Ignite over a free flame to constant weight. Record the loss in weight which represents the weight of silica.

6.5 Calculation

$$\text{Silicon, percent} = \frac{A \times 46{\cdot}72}{B}$$

where

A = weight in g of silica obtained, and

B = weight in g of the sample taken.

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